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TRANSLATOR'S DECLARATION

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, Kenji OHDAN, declare:

that I am thoroughly familiar with both the Japanese and English languages;

that the attached document represents a true full English translation of Japanese Patent Kokai 63-112429; and

That I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 19th day of December, 2000

Kenji Ohdan

TRANSLATOR

Tomikawa et al.

Translation of Japanese Patent Kokai 6-83063

(54) 【Title of the invention】 Chemical rays-polymerizable composition

(57) 【Abstract】

【Constitution】 The present invention is a chemical ray-polymerizable composition characterized by containing a silane compound represented by the general formula [I]

【Chem 1】 $(R_1O)_m-Si(R_3)_p-(OR_2)_n$. [I]

【Advantages】 According to the present invention, a chemical ray-polymerizable composition having photosensitivity suitable for the patterning works by the method of photolithography and characterized by excellent ultraviolet light transmissivity and heat resistance can be obtained.

【The claims】

【Claim 1】 A chemical ray-polymerizable composition characterized by containing

(a) a silane compound containing 30 to 100% by moles of a silane compound (A) represented by the general formula [I]

【Chem 1】 $(R_1O)_m-Si(R_3)_p-(CR_2)_n$ [I]

(in which R_1 is a benzyl group substituted by 1 to 4 of methoxy groups and/or nitro groups, R_2 is a monovalent hydrocarbon group of C1 to C6, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group, phenyl group or naphthyl group, R_3 is a monovalent hydrocarbon group of C1 to C12, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxypropyl group or epoxycyclohexylethyl group, m is an integer of 1 to 4, n is an integer of 0 to 3, p is an integer of 0 to 3 and m+n+p=4),

(b) water in an amount of 0.2 to 4 times by moles relative to the overall alkoxy groups in the said silane compound,

(c) an organic solvent in an amount of 0.1 to 10 times by weight relative to the said silane compound, and

(d) an acid or a base in 0.01 to 10% by moles relative to the said silane compound.

【Claim 2】 The chemical ray-polymerizable composition

characterized in that, in the silane compound in claim 1, a silane compound (B) represented by the general formula [II] is contained in a range not exceeding 70% by moles.

【Chem 2】 $(R_4O)_q-Si-(R_5)_r$ [II]

(in which R_4 is a monovalent hydrocarbon group of C1 to C6, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group or phenyl group, R_5 is a monovalent hydrocarbon group of C1 to C12, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxypropyl group or epoxy-cyclohexylethyl group, q is an integer of 1 to 4, r is an integer of 0 to 3 and q+r=4).

【Detailed description of the invention】

【0001】

【Field of industrial application】 The present invention relates to a chemical ray-polymerizable composition or, more particularly, relates to a silicon compound capable of pattern formation by a polymerization reaction proceeding by chemical rays such as ultraviolet light and the like.

【0002】

【Prior art technology】 In recent years, a demand is increasing in the field of semiconductors for EEPROM (Exposed Erasable Programmable Read Only Memory) in which the contents memorized by light are erasable. In this memory, the contents are rewritable by irradiating the memory chip with ultraviolet light of 254 nm. For this purpose, quartz glass is conventionally used as the window material but it is essential therefor that the package is formed of a ceramic. Ceramic packages, however, have a problem of extreme expensiveness not to allow a decrease in the cost of the products.

【0003】 On the other hand, while it is widely undertaken for many other products to prepare the packages of a plastic in order to accomplish a low cost of the product, it is practiced in this case to provide a thin coating of a heat-resistant resin such as a polyimide and the like on the surface of the silicon chip in order to decrease the thermal stress induced by the difference in the thermal expansion coefficient between the resin of the package and the silicon chip.

【0004】 While application of this technology to EEPROM is a due idea, no possibility is found when a product such as EEPROM has to be irradiated with ultraviolet light because polyimides heretofore used do not transmit ultraviolet light.

【0005】

【Problems to be solved by the invention】 Accordingly, the object of the present invention is to provide a chemical ray-polymerizable composition having good ultraviolet light transmissivity and heat resistance and suitable for patterning works by the method of photolithography.

【0006】

【Means to solve the problems】 Such an object of the present invention can be accomplished by taking following constitution.

【0007】 (1) A chemical ray-polymerizable composition characterized by containing

(a) a silane compound containing 30 to 100% by moles of a silane compound (A) represented by the general formula [I]

【Chem 3】 $(R_1O)_m-Si(R_3)_p-(OR_2)_n$ [I]

(in which R_1 is a benzyl group substituted by 1 to 4 of methoxy groups and/or nitro groups, R_2 is a monovalent hydrocarbon group of C1 to C6, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group, phenyl group or naphthyl group, R_3 is a monovalent hydrocarbon group of C1 to C12, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxypropyl group or epoxycyclohexylethyl group, m is an integer of 1 to 4, n is an integer of 0 to 3, p is an integer of 0 to 3 and m+n+p=4),

(b) water in an amount of 2 to 4 times by moles relative to the overall alkoxy groups in the said silane compound,

(c) an organic solvent in an amount of 0.1 to 10 times by weight relative to the said silane compound, and

(d) an acid or a base in 0.01 to 10% by moles relative to the said silane compound.

【0008】 (2) A chemical ray-polymerizable composition characterized in that, in the silane compound in (1) above, a silane compound (B) represented by the general formula [II] is contained in a range not exceeding 70% by moles.

【0009】

【Chem 4】

$(R_4O)_q\text{-Si-}(R_5)_r$ [II]

(in which R_4 is a monovalent hydrocarbon group of C1 to C6, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group or phenyl group, R_5 is a monovalent hydrocarbon group of C1 to C12, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxypropyl group or epoxycyclohexylethyl group, q is an integer of 1 to 4, r is an integer of 0 to 3 and q+r=4). The silane compound (A) in the present invention is represented by the general formula [I] given below.

【0010】

【Chem 5】 $(R_1O)_m\text{-Si}(R_3)_p\text{-}(OR_2)_n$. [I]

In the general formula [I], R_1 is a methoxy group and/or a benzyl group substituted for 1 to 4 by nitro groups. R_1O is an alkoxy group which is eliminated by light irradiation and forms a silanol group by being subjected to hydrolysis and has a characteristic that polymerization of the silane compound proceeds so as to be insolubilized in solvents by the dehydration condensation between the silanol groups formed by the hydrolysis. Such an alkoxy group (R_1O) includes nitrobenzylalkoxy groups, dinitrobenzylalkoxy groups, trinitrobenzylalkoxy groups, methoxybenzylalkoxy groups, dimethoxybenzylalkoxy groups, trimethoxybenzylalkoxy groups, nitromethoxybenzylalkoxy groups, dinitromethoxybenzylalkoxy groups, nitrodimethoxybenzylalkoxy groups and the like, though not limited thereto, but can be any alkoxy groups which can be eliminated by light. Particularly preferable among the above described are o-nitrobenzylalkoxy groups, dinitrobenzylalkoxy groups, methoxybenzylalkoxy groups and trimethoxybenzylalkoxy groups. And, m denotes an integer of 1 to 4 and, when m is 2 or larger, R_1s can be the same or different each from the others.

【0011】 In the above given general formula [I], R_2 denotes a monovalent C1-C6 hydrocarbon group, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group, phenyl group or naphthyl group. Preferable among

them are methyl group, ethyl group, 2-methoxyethyl group and 2-methoxypropyl group. And, n denotes an integer of 0 to 3 and, when n is 2 or larger, R_2s can be the same or different each from the others.

【0012】 R_3 denotes a monovalent C1-C12 hydrocarbon group, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxy-propyl group or epoxycyclohexylethyl group. Preferable among them are methyl group and phenyl group. And, p is an integer of 0 to 3 and m+n+p=4. When p is 2 or larger, R_3s can be the same or different each from the others.

【0013】 Particular examples of the silane compound (A) include silane compounds such as methyl dimethoxy nitrobenzylalk-oxysilanes, phenyl diethoxy methoxybenzylalkoxysilanes, ethyl dimethoxypropoxy dinitrobenzylalkoxysilanes, epoxy-cyclohexylethyldimethoxyethoxy dimethoxybenzylalkoxysilanes, glycidoxypropyl diphenoxy nitromethoxybenzylalkoxysilanes, glycidylmethoxyethoxy ethoxyethoxy trinitrobenzylalkoxysilanes, naphthylnaphthoxy butoxy dimethoxynitrobenzylalkoxysilanes, propylmethoxy ethoxy trimethoxybenzylalkoxysilanes, phenylethoxy bis(nitrobenzylalkoxy)silanes, methyl methoxy bis(methoxybenzylalkoxy)silanes, methyl tris(dinitrobenzyl-alkoxy)silanes, tetrakis(methoxybenzylalkoxy)silanes, nitrobenzylalkoxy methoxybenzylalkoxy dinitrobenzylalkoxy trimethoxybenzylalkoxysilanes, ethyl nitrobenzylalkoxy methoxybenzylalkoxy methoxysilanes, phenyl dinitrobenzylalkoxy methoxynitrobenzylalkoxy dimethoxybenzylalkoxysilanes, dimethylmethoxy nitrobenzylalkoxysilanes, diphenyl methoxy methoxybenzylalkoxysilanes, dimethyl bis(dinitrobenzyl-alkoxy)silanes, trimethyl nitrobenzylalkoxysilanes and the like, though not limited thereto. These can be used either singly or as a mixture of two kinds or more.

【0014】 It is important that the silane compound (A), which has practical photosensitivity and solubility behavior, is contained by 30 to 100% by moles in the overall silane compounds and it is more preferable that it is contained by at least 50% by moles. When smaller than 30% by moles, problems are caused that the sensitivity is decreased, solubility in solvents is decreased

or solubility is lost and so on.

【0015】 The silane compound (B) of the present invention represented by the general formula [II] is used together with the silane compound (A) and known silane compounds can be used adequately.

【0016】

【Chem 6】

$$(R_4O)_q\text{-Si-}(R_5)_r . \qquad [II]$$

In the general formula [II], R_4 denotes a monovalent C1 to C6 hydrocarbon group, 2-methoxyethyl group, 2-ethoxyethyl group, 2-methoxypropyl group, 2-ethoxypropyl group or phenyl group, of which methyl group, ethyl group, 2-methoxyethyl group and 2-methoxypropyl group are particularly preferable. And, q denotes an integer of 1 to 4 and, when q is 2 or larger, R_4s can be the same or different each from the others.

【0017】 R_5 denotes a monovalent C1 to C12 hydrocarbon group, phenyl group, naphthyl group, vinyl group, glycidyl group, glycidoxypropyl group or epoxycyclohexylethyl group, of which methyl group and phenyl group are particularly preferable. And, r denotes an integer of 0 to 3 with the proviso of q+r=4. When r is 2 or larger, R_5s can be the same or different each from the others.

【0018】 Typical examples of the silane compound (B) include tetramethyl alkoxy silanes, tetraethyl alkoxy silanes, tetrapropyl alkoxy silanes, methyl trimethoxy silane, methyl triethoxy silane, methyl tripropoxy silane, ethyl trimethoxy silane, ethyl triethoxy silane, phenyl trimethoxy silane, phenyl triethoxy silane, dimethyl dimethoxy silane, dimethyl diethoxy silane, methyl tris(2-methoxypropoxy) silane, methyl tris(2-methoxyethoxy) silane, vinyl trimethoxy silane, naphthyl triphenoxy silane, methyl triphenoxy silane, glycidyl trimethoxy silane, glycidoxypropyl triethoxy silane, epoxycyclohexylethyl triethoxy silane and the like, though not limitative thereto. Among them, alkyl trialkoxy silanes, of which r is 1, such as methyl triethoxy silane, phenyl trimethoxy silane and the like are particularly preferable in respect of solubility and others. It is preferable that the silane compound (B) is contained in

the overall silane compounds by 0 to 70% by moles or, more preferably, by 50% by moles or less.

【0019】 Water in the present invention is essential to effect hydrolysis of the alkoxy groups in the silane compounds to form silanol groups. It is important that the amount of use is, though theoretically equivalent to the total amount of the alkoxy groups in the silane compounds, practically, from 0.2 to 4 times by moles or, preferably, from 0.8 to 1.2 times by moles.

【0020】 The organic solvent used in the present invention is for dissolving the silane compound and water and, for example, alcoholic solvents such as methanol, ethanol, propanol, butanol and the like, glycol-based solvents such as ethyleneglycol monoethyl ether, ethyleneglycol monomethyl ether acetate, propyleneglycol monoethyl ether, diethyleneglycol diacetate and the like, ketone-based solvents such as methyl ethyl ketone, diisobutyl ketone, methyl isobutyl ketone and the like, bipolar aprotic solvents such as dimethylformamide, dimethyl acetamide, dimethyl sulfoxide, 1-methyl-2-pyrrolidone, γ-butyrolactone and the like, and so on can be used, of which the bipolar aprotic solvents are particularly preferable in respect of dissolving power. It is important that these organic solvents are used in 0.1 to 10 times by weight relative to the overall silane compounds. When the amount of use is smaller than 0.1 time by weight, gelation may eventually takes place while use in excess over 10 times by weight is undesirable because unevenness is sometimes caused in the coating film.

【0021】 The acid or base in the present invention acts as a catalyst for the hydrolysis of the above mentioned silane compounds with water. Usually, acids are preferably used as the catalyst in respect of the rate of hydrolysis and stability. As to the amount of addition thereof, it is important to add in 0.01 to 10% by moles or, preferably, in 0.1 to 5% by moles.

【0022】 As the acid, it is preferable to use hydrochloric acid, sulfuric acid, nitric acid, acetic acid, phosphoric acid, trifluoroacetic acid, methanesulfonic acid, monochloroacetic acid and the like. As the base, it is preferable to use amines such

as ammonia, methylamine, ethylamine, propylamine, dimethylamine, diethylamine, trimethylamine, triethylamine, tetramethylammonium hydroxide and the like. When the present invention is employed in the applications for the protective films, interlayer insulating films and the like of semiconductor devices, it is undesirable to use a base having an alkali metal such as sodium hydroxide, sodium carbonate and the like due to contamination of the semiconductor device.

【0023】 As the catalyst, furthermore, photoacid-generating agents capable of generating an acid by irradiation with light and photoamine-generating agents capable of generating an amine can be used either singly or in combination with other catalysts. The photoacid-generating agent is exemplified by benzoin tosylate, triphenylsulfonium triphrate, diphenyliodonium fluorophosphate and the like. As the photoamine-generating agent, reaction products of an isocyanate compound of an amine such as cyclohexylamine, hexamethylene diamine, ethylenediamine, diaminohexane, benzylamine, diaminodiphenyl ether, aniline and the like and nitrobenzyl alcohol or methoxybenzyl alcohol can be used.

【0024】 In order to further improve the photosensitivity behavior of the present invention, it is optional to admix a sensitizer such as Michler's ketone and the like in 1 to 30% by weight relative to the overall silane compounds.

【0025】 The photosensitive solution is obtained by mixing the above described ingredients.

【0026】 In the following, a description is given of the using method of the solution of the composition of the present invention.

【0027】 Firstly, a substrate such as a silicon wafer, ceramic plate and the like is coated with the solution by the method of the spin coating method, spray method and the like. In the next place, heat is added for evaporating unnecessary solvent to form a film. The temperature which can be used in this step is preferably 120 ℃ or below or, more preferably, 100 ℃ or below because, if the temperature here is higher than 120 ℃ ,

the alkoxy groups are also hydrolyzed resulting in eventual disappearance of the photosensitivity. Thereafter, a light exposure treatment is undertaken. The light exposure can be performed by an instrument such as aligners, steppers and the like currently under preferable use in the semiconductor industry. Since the sensitivity to long wavelength is low in the present invention, it is preferable to employ the i-line (365 nm) of a high-pressure mercury lamp, excimer lasers (308 nm and 248 nm) and the like but, being not limited to such a light of a single specific wavelength, continuous lights and those as a mixture of a plurality of wavelengths can also be used satisfactorily.

【0028】 Light exposure is followed by second baking at a temperature of about 100 ℃ so as to facilitate proceeding of the dehydration condensation of the silanol groups formed by the light. Also in this case, it is preferable that the baking is conducted at a temperature not exceeding 120 ℃ because, when conducted at a temperature exceeding 120 ℃, residual alkoxy groups in the unexposed portions are dissociated by the heat to cause dehydration condensation. It is possible in many cases to omit this step.

【0029】 Thereafter, development is undertaken. Preferably usable as the development liquid include bipolar aprotic solvents such as N,N-dimethylformamide, N-methyl-2-pyrrolidone, dimethylsulfoxide, γ-butyrolactone, dimethylacetamide, sulfolane and the like, alcohols such as methanol, ethanol, propanol, butanol, ethyleneglycol, propyleneglycol, ethyleneglycol monoethyl ether, propyleneglycol monomethyl ether and the like, ethers such as tetrahydrofuran, dioxane and the like and ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone, diisopropyl ketone, cyclohexanone and the like as well as mixtures thereof. Aqueous solutions of an organic amine such as tetramethylammonium hydroxide and the like, and so on can also be used.

【0030】 The development method with them includes the method of dipping in these development liquids, the method of spraying with rotation, the method of mounting the liquid on the

substrate and so on and an adequate development method can be selected in the present invention depending on the condition.

〖0031〗 The development is followed by the addition of a still higher temperature so as to react all of the silanol groups to give a complete polysiloxane resin. In this case, the preferable temperature is 200 ℃ or higher or, more preferably, from 250 ℃ to 500 ℃. When this temperature is lower than 200 ℃, unreacted silanol groups remain possibly to adversely influence on adhesion and electric properties. And, when in excess over 500 ℃ or higher and, particularly, when the film has a thickness of 3 μm or larger, cracks are undesirably formed in the film eventually.

〖0032〗 The chemical ray-polymerizable composition of the present invention can naturally be used in the application fields of photosensitive resins such as, besides the protective film of EEPROM, photoresists, interlayer insulating films of semiconductor devices and substrates, protective films of line sensors, protective films of color filters and the like.

〖0033〗

〖Examples〗 In the following, the present invention is described in particular by making reference to Examples.

〖0034〗 <Synthesis of a methyl dimethoxy-o-nitrobenzyl alkoxy silane> 22 g of methyl triacetoxy silane were dissolved in 30 ml of tetrahydrofuran and a solution of 15.3 g of o-nitrobenzyl alcohol dissolved in 30 ml of tetrahydrofuran was added thereto dropwise through a dropping funnel taking 10 minutes. This solution was reacted at 30 ℃ for 2 hours and then 6.4 g of methanol were added dropwise taking 3 minutes. Thereafter, reaction was undertaken at 30 ℃ for 1 hour and further reaction was undertaken at 40 ℃ for 2 hours. This solution was subjected to reduced-pressure distillation under a reduced pressure of 10 mmHg with a vacuum pump to give a methyl dimethoxy-o-nitrobenzyl alkoxy silane as a fraction having a boiling point of 180 ℃ to 185 ℃.

〖0035〗 <Synthesis of a methyl dimethoxy-p-methoxybenzyl alkoxy silane> 22 g of methyl triacetoxy silane were dissolved in 30 ml of tetrahydrofuran and a solution of 13.8 g of p-methoxy-

benzyl alcohol dissolved in 30 ml of tetrahydrofuran was added thereto dropwise through a dropping funnel taking 10 minutes. A little heat generation was observed in this time. This solution was reacted at 30 ℃ for 2 hours and then 6.4 g of methanol were added dropwise taking 3 minutes. Thereafter, reaction was undertaken at 30 ℃ for 1 hour and further reaction was undertaken at 40 ℃ for 2 hours. This solution was subjected to reduced-pressure distillation under a reduced pressure of 10 mmHg with a vacuum pump to give a methyl dimethoxy-p-methoxybenzyl alkoxy silane as a fraction having a boiling point of 170 ℃ to 180 ℃ .

【0036】 Example 1

7.74 g (50% by moles) of the methyl dimethoxy-o-nitrobenzyl alkoxy silane, 2.72 g (33% by moles) of methyl trimethoxy silane and 1.98 g (17% by moles) of phenyl trimethoxy silane were added to 3.24 g of water, 2.18 g of N-methyl-2-pyrrolidone and 0.2 g of phosphoric acid and agitated vigorously. While with slight heat evolution at the initial stage, the solution soon reaches a stationary state. This solution was subjected to a reaction at 50 ℃ for 4 hours to give a photosensitive solution.

【0037】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃ . Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0038】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development for 2 minutes with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that a pattern remained with an exposure dose of 300 mJ/cm^2 or more to exhibit photosensitive property.

【0039】 Example 2

7.44 g (50% by moles) of the methyl dimethoxy-p-methoxybenzyl alkoxy silane, 2.72 g (33% by moles) of methyl trimethoxy silane and 1.98 g (17% by moles) of phenyl trimethoxy silane were added to 3.24 g of water, 2.18 g of N-methyl-2-pyrrolidone

and 0.2 g of acetic acid and agitated vigorously. While with slight heat evolution at the initial stage, the solution soon reaches a stationary state. This solution was subjected to a reaction at 50 ℃ for 4 hours to give a photosensitive solution.

【0040】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0041】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development for 2 minutes with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that a pattern remained with an exposure dose of 500 mJ/cm^2 or more to exhibit photosensitive property.

【0042】 Example 3

A photosensitive solution was prepared by further addition of 0.1 g of triphenylsulfonium triphrate to the formulation of the photosensitive solution in Example 1.

【0043】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0044】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development for 2 minutes with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that a pattern remained with an exposure dose of 200 mJ/cm^2 or more to exhibit photosensitive property.

【0045】 Example 4

A photosensitive solution was prepared by further addition of 0.1 g of benzoin tosylate to the formulation of the photosensitive solution in Example 2.

【0046】 A silicon wafer was spin-coated with the above mentioned

photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0047】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development for 2 minutes with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that a pattern remained with an exposure dose of 250 mJ/cm^2 or more to exhibit photosensitive property.

【0048】 Example 5

12.85 g (100% by moles) of the methyl dimethoxy-o-nitrobenzyl alkoxy silane were added to 2.70 g of water, 1.82 g of N-methyl-2-pyrrolidone and 0.23 g of phosphoric acid and agitated vigorously. While with slight heat evolution at the initial stage, the solution soon reaches a stationary state. This solution was subjected to a reaction at 50 ℃ for 4 hours to give a photosensitive solution.

【0049】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0050】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development for 2 minutes with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that a pattern remained with an exposure dose of 150 mJ/cm^2 to exhibit photosensitive property.

【0051】 Comparative Example 1

2.58 g (20% by moles) of the methyl dimethoxy-o-nitrobenzyl alkoxy silane, 4.08 g (60% by moles) of methyl trimethoxy silane and 1.98 g (20% by moles) of phenyl trimethoxy silane were added to 3.24 g of water, 2.18 g of N-methyl-2-pyrrolidone and 0.2 g of phosphoric acid and agitated vigorously. While with slight

heat evolution at the initial stage, the solution soon reaches a stationary state. This solution was subjected to a reaction at 50 ℃ for 4 hours to give a photosensitive solution.

【0052】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0053】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that no dissolution could be accomplished even by dipping in the developer solution for 30 minutes not to exhibit photosensitive property.

【0054】 Comparative Example 2

4.08 g (75% by moles) of methyl trimethoxy silane and 1.98 g (25% by moles) of phenyl trimethoxy silane were added to 3.24 g of water, 2.18 g of N-methyl-2-pyrrolidone and 0.2 g of hydrochloric acid and agitated vigorously. While with slight heat evolution at the initial stage, the solution soon reaches a stationary state. This solution was subjected to a reaction at 50 ℃ for 4 hours to give a photosensitive solution.

【0055】 A silicon wafer was spin-coated with the above mentioned photosensitive solution and subjected to a baking treatment for 3 minutes on a hot plate at 80 ℃. Subsequently, light exposure of 1 J/cm^2 (365 nm) was conducted through a grey scale mask by using a contact aligner manufactured by Canon.

【0056】 The same was subjected to another baking treatment at 100 ℃ for 1 minute and subjected to dipping development with a developer solution consisting of a composition of 60 parts by weight of N-methyl-2-pyrrolidone and 40 parts by weight of xylene. By conducting rinse with isopropanol, thereafter, it was found that no dissolution could be accomplished even by dipping in the developer solution for 30 minutes not to exhibit photosensitive property.

【0057】

【Advantages of the invention】 According to the present invention, a chemical ray-polymerizable composition with excellent ultraviolet light transmissivity and heat resistance having photosensitivity suitable for patterning works by the procedure of photolithography can be obtained.